DEPARTMENT OF AGRICULTURE,
CEYLON.

BULLETIN No. 32.

VULCANIZATION TESTS.

Investigations at the Imperial Institute on Samples of Plantation Para Rubber from Ceylon prepared in connection with the Rubber Research Scheme.

Fifth Interim Report.

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VULCANIZATION TESTS.

Investigations at the Imperial Institute on Samples of Plantation Para Rubber from Ceylon prepared in connection with the Rubber Research Scheme.

FIFTH INTERIM REPORT.

THE present report deals with a number of specimens of rubber prepared by Mr. L. E. Campbell, B.Sc., the Rubber Research Chemist, in order to determine the effect of diluting the latex to varying degrees before coagulating. The specimens, which number twenty-six, belong to the Intermediate Series I. (P 1 to P 8) and to Sections I., II., and III. of Series III. The exact method of preparation of the specimens is given in the following account.

INTERMEDIATE SERIES I.

Dilution of Latex.—Rubber prepared in sheet. The experiments were made with latex from the seven-year old trees used for Series I.

Separate portions of the latex, which contained 33 per cent. of dry rubber, were diluted with gradually increasing quantities of water. The same amount of acetic acid was used for coagulation in each case, viz., 0·206 gram of pure acetic acid per 100 cc. of undiluted latex. The acid after dilution was stirred into the latex, except in P 7 and P 8, where the diluted acid was simply poured into the latex. The following table gives particulars of the specimens :—

Serial No.	Volume of Latex taken.	Volume of Water added.	Volume of diluted Acid added.	Final Dilution. Volume to which 1 Volume of Latex was diluted.	Time required for Coagulation.	Time of drying of Rubber.
	Litres.	Litres.	Litres.			Weeks.
P 1	2	Nil	0·25	1·125	Immediate	3
P 2	2	Nil	1·0	1·5	1 to 2 minutes	3
P 3	2	0·5	0·5	1·5	do.	3
P 4	2	1·0	1·0	2·0	Thickened in 2 minutes; hardened gradually	3
P 5	2	3·0	1·0	3·0	Appreciably slower than in P 4	3
P 6	2	9·0	1·0	6·0	About 30 minutes	Not stated
P 7*	1	4·5	0·5	6·0	do.	
P 8	2	11·0	1·0	7·0	45 minutes	

* Two samples prepared.

All these specimens consisted of sheet rubber prepared in the usual manner.

SERIES III.

Experiments conducted with Latex from the Trees 16 to 20 Years old used for Series II.

Section I.—Dilution of Latex.

Rubber prepared in sheet.

Date of experiment: June 4, 1914.

Rainfall: 0·79 inch.

Percentage of dry rubber in latex: 26 per cent.

This section includes a range of dilutions up to 1 volume of latex with 8 volumes of water. In this case the coagulation was not carried out by the standard method, but was brought about by adding 40 cc. of 10 per cent. acetic acid to 3 litres of latex containing 26 per cent. of dry rubber. Particulars of the specimens are given in the following table :—

Serial No.	Volume of Latex taken.	Volume of Water added.	Volume of Acetic Acid (10 per Cent.) added.	Final Dilution. Volume to which 1 Volume of Latex was diluted.	Time of drying of Rubber.
	Litres.	Litres.	Litres.		Weeks.
186	3	Nil	0·04	1·013	5
187	3	0·04	0·04	1·027	5
188			No sample.		
189	3	0·375	0·04	1·138	3
190	3	1·5	0·04	1·513	3
191	3	3·0	0·04	2·013	3
192	3	6·0	0·04	3·013	3
193	3	15·0	0·04	6·013	5
194	3	24·0	0·04	9·013	8

All the samples were made into sheet in the usual manner.

With reference to the time required for coagulation in these specimens, Mr. Campbell supplied the following note :—

"The apparent difference in the rates of coagulation would appear to depend, not so much on an actual difference in the rates at which the emulsoid phase of the latex is destroyed, as on the rates at which the minute particles of rubber become coherent one with the other. To take an example: in the cases of Nos. 186, 187, and 188 the latex commenced to 'thicken' in about 15 minutes, and a solid coherent coagulum was formed in 40 minutes. In the cases of Nos. 193 and 194, on the other hand, 2 or 3 hours elapsed before a solid coherent coagulum was formed similar to that produced in 40 minutes in the cases of Nos. 186, 187, and 188. This proved to be not

due principally to a slower rate of destruction of the emulsoid state, for after 15 minutes the latex had changed entirely in character: the solid particles of rubber had formed, but in the form of very small flakes, which took a very considerable time to cohere. Microscopic examination showed that Brownian movement of the globules had ceased. The apparent difference in rates of coagulation must depend on the rates at which the particles of rubber cohere together to form a coagulum."

Section II.—Dilution of Latex.

Rubber prepared in sheet.

Date of experiment: June 7, 1914.

Rainfall: 0·06 inch.

Percentage of dry rubber in latex: 34 per cent.

The samples were prepared as follows:—

Serial No.	Volume of Latex taken.	Volume of diluted Acid added.	Final Dilution. Volume to which 1 Volume of Latex was diluted.	Time of drying of Rubber.
	Litres.	Litres.		Weeks.
195	2·0	0·1	1·05	4
196*	2·0	2·0	2·0	3
197	2·0	4·0	3·0	4
198	2·0	12·0	7·0	8

* Control sample.

All the samples were made into sheet in the usual manner.

Section III.—Dilution of Latex.

Rubber made into crêpe.

Date of experiment: June 16, 1914.

Rainfall: 0·47 inch.

Percentage of dry rubber in latex: 32 per cent.

The samples were prepared as follows:—

Serial No.	Volume of Latex taken.	Volume of diluted Acid added.	Final Dilution. Volume to which 1 Volume of Latex was diluted.	Time of drying of Rubber.
	Litres.	Litres.		Days.
199	4·0	0·2	1·05	10
200	4·0	4·0	2·0	10
201	4·0	8·0	3·0	10
202	4·0	24·0	7·0	10

All the samples in this section were crêped in exactly the same way: seven times through rough rollers and once through smooth rollers.

RESULTS OF EXAMINATION.

(1) Vulcanization and Mechanical Tests.

The specimens have been vulcanized and submitted to mechanical tests in exactly the same manner as the previous samples, and the results obtained are given in the following table :—

INTERMEDIATE SERIES.

Dilution of Latex.

Sheet Rubber.

(For particulars see page 1.)

	Volumes.	Serial No.	Time of Cure. Minutes at 50 lb. Pressure.	Tensile Strength. Lb. per Sq. Inch.	Elongation at Breaking Point. Per Cent.	Permanent Set Elongation. Per Cent.
1 volume of latex was diluted to	1·125	P 1	60	2,670	855	2·06
	1·5	P 2	62	2,370	876	2·28
	1·5	P 3	62	2,440	854	2·28
	2·0	P 4	62	2,650	894	2·33
	3·0	P 5	65	2,400	902	2·76
	6·0	P 6	62	2,470	882	2·06
	6·0	P 7	62	2,520	869	2·06
	7·0	P 8	65*	2,230	883	2·80

* Slightly under-cured.

SERIES III.

SECTION I.

Dilution of Latex.

Sheet Rubber.

(For particulars see page 2.)

	Volumes.					
1 volume of latex was diluted to	1·0013	186	70	2,660	865	2·00
	1·0027	187	70	2,510	869	1·98
	—	188	No sample.			
	1·138	189	80	2,530	847	1·92
	1·513	190	70	2,560	872	2·13
	2·013	191	80	2,370	881	2·11
	3·013	192	78	2,300	862	2·55
	6·013	193	80	2,490	856	2·30
	9·013	194	80	2,380	862	2·24

SECTION II.

Dilution of Latex.

Sheet Rubber.

(For particulars see page 3.)

	Volumes.					
1 volume of latex was diluted to	1·05	195	70	2,470	866	2·26
	2·0	196†	80	2,560	871	2·12
	3·0	197	80	2,360	889	2·27
	7·0	198	70	2,560	863	1·98

† Control sample.

SECTION III.

Dilution of Latex.

Crêpe Rubber.

(For particulars see page 3.)

	Volumes.	Serial No.	Time of Cure. Minutes at 50 lb. Pressure.	Tensile Strength. Lb. per Sq. Inch.	Elongation at Breaking Point. Per Cent.	Permanent Set Elongation. Per Cent.
1 volume of latex was diluted to	1·05	199	100	2,550	872	1·71
	2·0	200	110*	2,450	837	2·06
	3·0	201	110	2,220	881	2·89
	7·0	202	110	2,290	875	2·06
Control		224	80	2,320	867	2·54

* Slightly over-cured.

(2) Chemical Composition.

The whole of the specimens have been submitted to analysis in order to determine whether the dilution of the latex produces any effect on the composition of the rubber. The results are given in the following table :—

INTERMEDIATE SERIES.

Dilution of Latex.

Sheet Rubber.

(For particulars see page 1.)

	Volumes.	Serial No.	Washing Loss.	Composition of dry washed Rubber. Ash.	Resin.	Protein.	Caoutchouc.
			Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.
1 volume of latex was diluted to	1·125	P 1	0·50	0·29	2·92	2·50	94·29
	1·5	P 2	0·35	0·24	2·84	2·41	94·51
	1·5	P 3	0·50	0·24	2·80	2·35	94·61
	2·0	P 4	0·35	0·23	2·70	2·35	94·72
	3·0	P 5	0·40	0·25	2·51	2·32	94·92
	6·0	P 6	0·20	0·18	2·78	2·28	94·76
	6·0	P 7	0·40	0·20	2·76	2·19	94·85
	7·0	P 8	0·30	0·15	2·67	2·33	94·85

SERIES III.

SECTION I.

Dilution of Latex.

Sheet Rubber.

(For particulars see page 2.)

	Volumes.	Serial No.	Washing Loss.	Composition of dry washed Rubber. Ash.	Resin.	Protein.	Caoutchouc.
			Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.
1 volume of latex was diluted to	1·013	186	0·60	0·26	2·06	2·08	95·60
	1·027	187	0·55	0·29	2·13	1·85	95·73
	—	188			No sample.		
	1·138	189	0·60	0·23	2·05	2·10	95·62
	1·513	190	0·60	0·18	1·99	1·96	95·87
	2·013	191	0·30	0·24	2·24	2·07	95·45
	3·013	192	0·40	0·18	2·33	1·85	95·64
	6·013	193	0·50	0·21	2·12	2·06	95·61
	9·013	194	0·30	0·19	2·28	2·01	95·52

SECTION II.

Dilution of Latex.

Sheet Rubber.

(For particulars see page 3.)

	Volumes.	Serial No.	Washing Loss.	Composition of dry washed Rubber. Ash.	Resin.	Protein.	Caoutchouc.
			Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.
1 volume of latex was diluted to	1·05	195	0·60	0·26	2·01	2·07	95·66
	2·0	196	0·40	0·19	1·71	1·92	96·18
	3·0	197	0·40	0·16	1·57	1·86	96·41
	7·0	198	0·40	0·12	2·30	1·77	95·81

SECTION III.

Dilution of Latex.

Crêpe Rubber.

(For particulars see page 3.)

	Volumes.						
1 volume of latex was diluted to	1·05	199	0·60	0·31	2·79	2·42	94·48
	2·0	200	0·35	0·21	2·57	2·34	94·88
	3·0	201	0·35	0·22	2·56	2·31	94·91
	7·0	202	Sample too small for analysis.				
Control		224	0·50	0·24	2·23	2·70	94·83

Remarks.

INTERMEDIATE SERIES.

In this set of samples (P 1 to P 8) there does not appear to be any definite connection between the degree of dilution of the latex and the time of cure or the quality of the rubber as judged by the results of the vulcanization tests. The variations in the time of cure, tensile strength, elongation, and permanent set are comparatively small, and show no regular relation to the increasing dilution of the latex.

The time of cure varied from 60 to 65 minutes, which is a little below the average of the control sheet specimens previously examined; P 1, which was made from the least diluted latex, had the shortest time of cure, but the differences in the series are only slight. All the samples, with the exception of P 8, which was slightly under-cured, gave good tensile strengths, ranging from 2,370 to 2,670 lb. per square inch; P 1 and P 4 (dilution 1·125 and 2·0 respectively) gave the highest figures, and were practically equal in respect of tensile strength.

With regard to the chemical composition of these samples, the increased dilution of the latex appears to have caused a fairly steady decrease in the amounts of ash, protein, and

resin present in the rubber, with a corresponding increase in the percentage of caoutchouc. This result is probably due to the larger bulk of liquid retaining more of the non-caoutchouc constituents in solution.

Series III.

In Section I. the time of cure varied from 70 to 80 minutes; three of the samples prepared from the least diluted latex, Nos. 186, 187, and 190, all cured in 70 minutes, but No. 189 took 80 minutes, like the samples Nos. 191 to 194 prepared from more diluted latex. The tensile strength was good throughout the section, the figures ranging from 2,300 to 2,660 lb. per square inch. Three of the samples prepared from the more diluted latex, Nos. 191, 192, and 194, were a little inferior in mechanical properties to Nos. 186 to 190. No definite conclusion can be drawn, however, on this point, as No. 193 (final dilution 6·0) gave very good results, and was approximately equal to No. 187, which was made from practically undiluted latex.

There does not appear to be any connection in the samples of Section I. between the composition of the rubber and the degree of dilution such as was evident in the Intermediate Series P 1 to P 8.

The four samples of Section II. cured in 70 to 80 minutes, the specimens from both the lowest and the highest dilution curing in the same time (viz., 70 minutes), whilst the intermediate specimens required 80 minutes. All four samples had good tensile strength, the values ranging from 2,360 to 2,560 lb. per square inch. No. 197 (dilution 3·0) had a somewhat lower tensile strength than the three other samples. Sample No. 198 (dilution 7·0) gave almost identical figures in the mechanical tests to No. 196 (dilution 2·0), which served as the control sample for the section.

In Section II. there is a steady decrease in the amounts of ash, resin, and protein present in the rubber, with increase of dilution of the latex, except that in No. 198 the percentage of resin shows an unaccountable increase.

The samples in Section III. were in the form of crêpe, and therefore required a longer time of cure than the control sheet. The time of cure of the crêpe samples varied from 100 to 110 minutes, compared with 80 minutes for the control sheet; the

sample of lowest dilution cured in the shortest time (100 minutes), and the other three samples (dilutions 2·0, 3·0, and 7·0 respectively) all required 110 minutes. The two samples of lowest dilution gave much better tensile results than the two of higher dilutions, and were in fact better in this respect than the control sample No. 224.

In composition the samples Nos. 199–201 show a slight decrease in ash, resin, and protein with increasing dilution of the latex, but unfortunately the amount of No. 202, the highest dilution, was not sufficient for both analysis and mechanical tests.

General Conclusions.

The results of these experiments to determine the effect of dilution of the latex on the vulcanizing and mechanical properties of the rubber are somewhat inconclusive.

The effect of dilution on the rate of cure is not very marked. The samples prepared from undiluted or slightly diluted latex have in each case given good results in the mechanical tests, whereas those prepared from much diluted latex are in some cases distinctly lower.

The results are too irregular to allow of the conclusion that excessive dilution of the latex has a deleterious influence on the qualities of the rubber, although this appears to be so in some cases. In other instances, however, this was not the case, whilst the diminution effected by dilution in the quantities of mineral constituents, protein, and resin may be of importance. It is desirable that further experiments should be made with a view to clearing up the discrepancies disclosed by this report.

March 9, 1917.